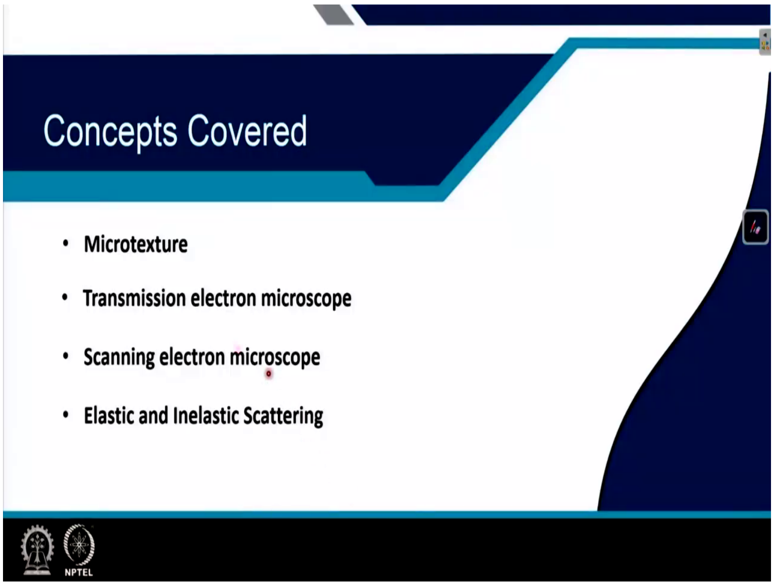
Texture in Materials

Prof. Somjeet Biswas
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Kharagpur

Module - 06
Micro texture measurements using EBSD technique in SEM
Lecture - 28
Basics of Electron Microscopy - I

Good afternoon everyone and today we will be starting Module 6 which is Micro texture measurement using EBSD technique in Scanning Electron Microscope. This is Lecture Number 28 where we will start with a little understanding of basically the prerequisite of the course, but a little understanding on the Basics of Electron Microscopy.

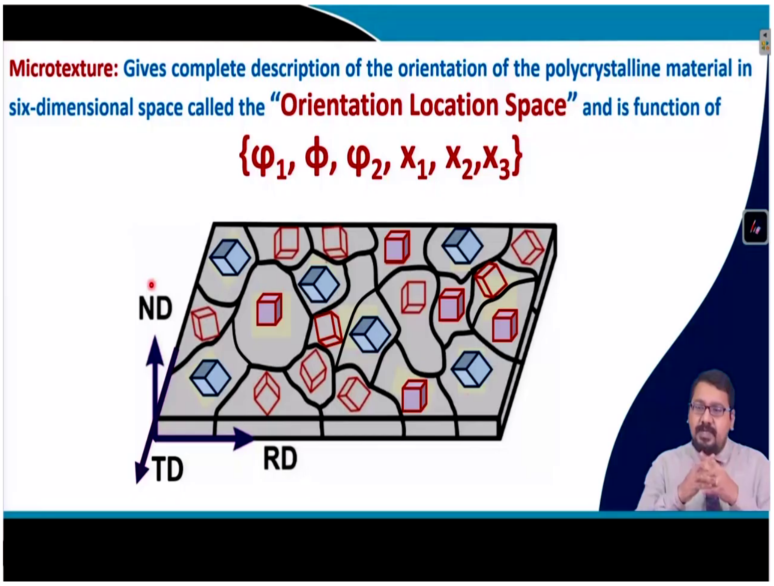
(Refer Slide Time: 01:05)



The concepts that we will be covered in this lecture are; what is micro texture and we know about this a little bit we have seen this. What is transmission electron microscope a little bit, what kind of radiations that comes and how we detect those radiations. Scanning electron microscopy or a little bit of it similar things what kind of radiations comes out of it and how we detect those.

We will mainly focus on elastic and inelastic scattering of electrons while we cover this transmission and scanning electron microscopies.

(Refer Slide Time: 01:51)



So, let us give what is micro texture the definition of it and we have defined it earlier also, but let us define it again here. It gives the complete description of the orientation of the poly crystalline material in six dimensional space basically the micro texture and the macro texture does not give this information. How it is given, it gives the information of the "Orientation Location Space".

So, it can give information of phi 1, phi, phi 2 and it can give information of the X Y and Z of the sample X 1, X 2 and X 3 of the sample. So, the position where the information is coming from. So, if we look in the pointer you will see that while we have given this kind of a schematic image of a polycrystalline material having so many grains and we have shown the the orientation of these grains each of these grains in terms of positioning its unit cell over it ok.

This is a schematic we have shown that this is RD TD and ND. So, if we are putting a sample a poly crystalline sample like this under a TEM or an SEM to obtain this kind of micro texture information that is in to obtain kikuchi patterns basically we will come to it later. So, what we do is that the electron beam basically falls on a very small portion of the sample it falls on a very small portion of the sample like the size of the beam would be in few nanometers and it falls on the portion a small portion of the sample a small portion like the pointer here.

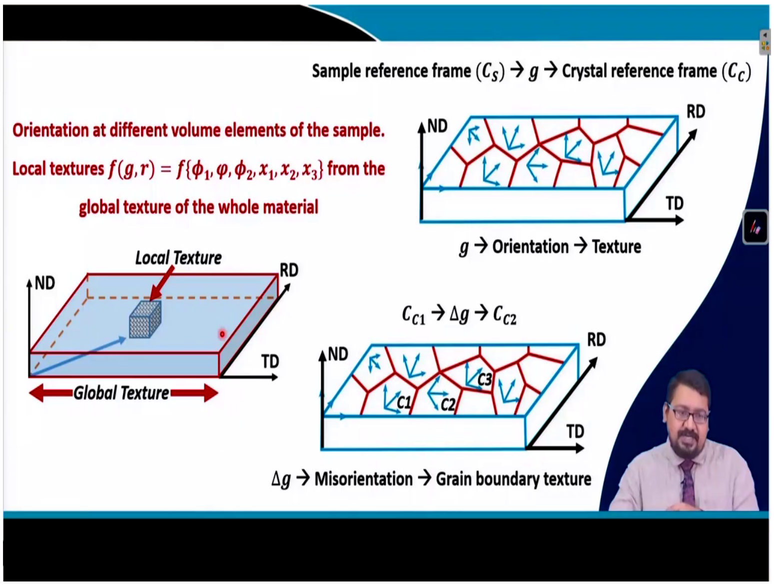
Just of the size of this pointer and then it obtains diffraction information from it in form of kikuchi pattern, from only that area where the pointer is sitting now and that kikuchi patterns information is can be converted into phi 1, phi, phi 2 because the kikuchi pattern has a relationship with respect to the sample reference direction. That means, that when we are putting the sample in the SEM we should put it in such a way that we should know which is the direction of RD, which is the direction of TD and of course, ND is above right if we are measuring the ND plane.

So, by that we obtain the information of that this small area and then we raster. So, we go one-step ahead and do the same thing and then one step ahead and do the same thing. So, we keep on measuring this multiple number of times throughout the whole sample we raster the whole sample and take information of the kikuchi pattern and we raster it like this and we go like this and we go and we scan the whole sample.

The part of the sample which we basically scan a small area of the sample can be scanned in this way you cannot scan a very large area of the sample and that is why it is it gives the information of the texture in a micro scale. So, it is micro textured it does it may not be a full representative of the sample, but it gives the full information of that particular position where the electron beam is falling and then it is rastering.

Now, various softwares and techniques have been established to keep information of the kikuchi pattern converting it into phi 1, phi, phi 2 information of the X 1 and X 2. Say for example, the horizontal along RD is X 1 and the along TD is X 2 and at the same time you must be wondering where this X 3 is coming from right. So, now, a days with the invention of ion milling techniques and with the integration of ion beam microscopy with the electron microscopy. So, ion beam is used to mill a part of the sample. So, that it reveals a surface below this surface and therefore, one can obtain three dimensional information of phi 1, phi, phi 2 ok and we will show this here.

(Refer Slide Time: 06:59)



So, what I meant to say if you look into this figure as I said that the information of the texture in case of micro texture is phi 1, phi, phi 2 x 1, x 2, x 3. Using a iron milling technique one can get the information of the surface and then one can go below say 1 micrometer or 10 micrometer and take another information from that surface and then below in x 3 that is along ND.

So, micro texture not only gives the information of the texture of that particular area or volume, but it also gives. So, if we take the texture of that volume that is g that is orientation we can get the information of g with respect to means the orientation of the crystal with respect to the sample direction we have talked about this earlier and in case of micro texture we can also get the information of the misorientations right. So, if C 1 is one crystal or one grain and C 2 is another grain so, the relationship between C 1 and C 2 that is delta g that is the misorientation angle and the axis could be obtained. So, this is known as grain boundary texture right. So, on the other hand we can also get the information of the sub grain boundaries of course, depending upon the resolution of the microscope for example, in case of tem it is 0.1 degrees and in case of SEM EBSD it is 2 degrees I am telling you earlier.

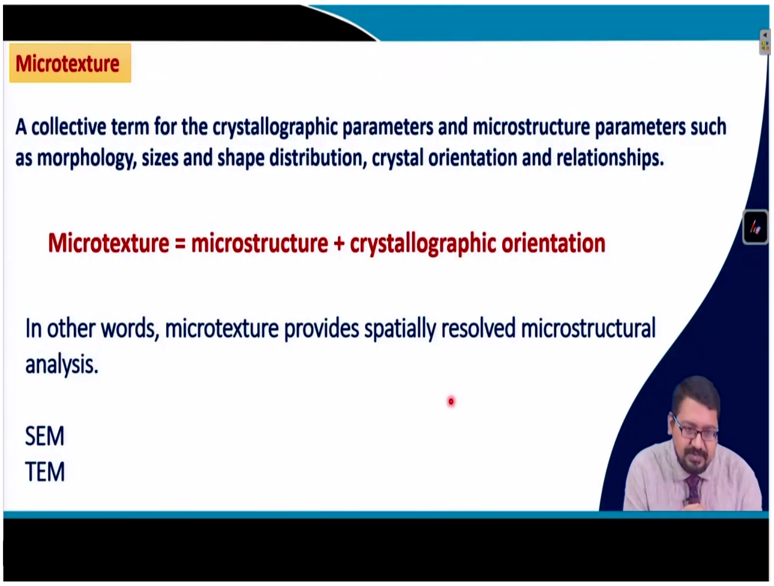
Now, if we have any misorientation which is greater than 2 degrees we can; obviously, obtain it using this micro texture operation and then therefore, we can get the information of the geometrically necessary boundaries which are also known as low angle boundaries or very low angle boundaries in case of texture evolution and in texture community basically.

So, very low angle boundaries are basically 2 to 5 degree misorientation angle boundaries and low angle boundaries are basically considered 5 to 15 degree misorientation boundaries and both of them are geometrically necessary boundaries of course, because they create a misorientation a orientation difference between two positions of the grains which is separated by this particular misorientation boundary.

And of course, it gives the information of the grain boundary because grain boundaries are actually high angle grain boundaries greater than 15 degrees are known as high angle grain boundaries. Here also there are divisions where the high angle grain boundaries could be random and or the high angle boundaries are specific like the specific boundaries like special boundaries right.

Like we call it coincidence site lattice boundaries it has a specific angle and a specific axis and its energy is quite lower than the energy of any random high angle boundaries and because the it has few of its atoms or bases in coincident, so that it has some kind of periodicity in it. So, an increased periodicity reduces the energy of that coincidence site lattice boundaries or special boundaries. And all these can be detected while we do a micro texture measurement and it could not be obtained while we are carrying out macro texture measurement in an xRD.

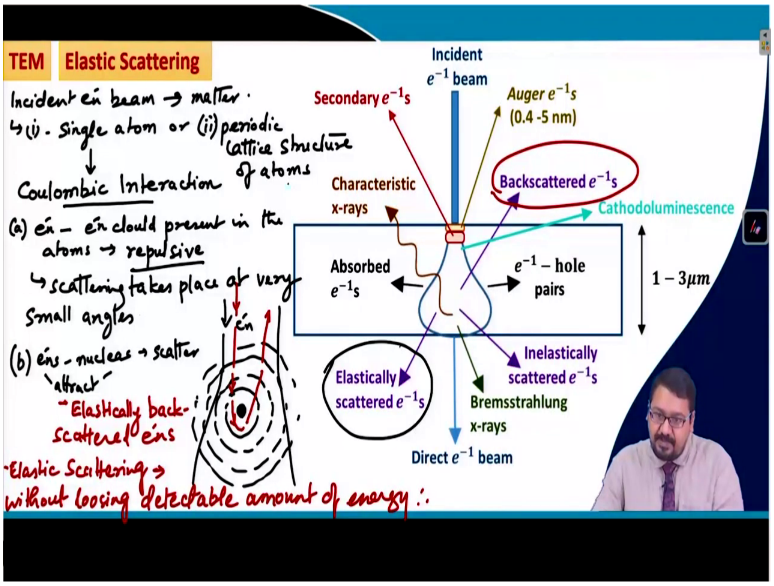
(Refer Slide Time: 10:53)



So, what is micro texture as a whole? It is the collective information of all the crystallographic parameters and the microstructural parameters such as morphology, size, shape distribution, crystal orientation, misorientation, their relationship, coincidence site, lattice boundary everything.

So, micro texture is basically microstructure plus crystallographic orientation. In other words micro texture gives us spatially resolved microstructural information quantitative microstructural information including its crystallographic information or orientation information and this can be done using scanning electron microscope or transmission electron microscope right.

(Refer Slide Time: 11:45)



So, let us go into transmission electron microscope and see what are the different kinds of radiations that can be generated from it; while an incident electron beam basically falls over it. You can see that in a transmission electron microscope the specimen thickness is actually very thin right.

It is thin such that the when the electron beam is falling the interaction volume of that falling electron beam or the falling electron or the striking electron on the surface of the sample is more or less equal or higher than the thickness of the sample. So, the thickness of the sample is usually in a range of 1 to 3 micrometer like that. So, it is a very thin sample which is put on a copper grid in a TEM and then it is basically radiated by an electron beam.

So, what are the radiation that comes up while the electron beam falls on it? You can see that Auger electrons comes out, secondary electrons comes out, characteristics x-rays comes out, back scattered electrons comes out, elastically scattered electrons comes out, inelastically scattered electrons comes out, white radiation or bremsstrahlung x-rays comes out. Definitely there is a direct beam because the beam intensity is so high that there will be some direct beams and there will be electron hole pair formation and cathode illuminations.

Now, let us take one by one how the various kinds of radiations or scattering takes place and let us take elastic scattering initially. Now when the incident electron beam interacts with this matter, which is poly crystalline matter and it we can consider it that it is interacting with a single atom or we can consider that it is interacting with a periodic lattice structure right structure of atom right.

So, if we consider that the incident beam is interacting with a single atom. So, this interaction is known as coulombic interaction. And if you look what is coulombic interaction? So, if electron that is falling that is from the incident beam interacts with electron cloud of these conductive poly crystalline metallic samples which is present in the atoms; then due to their repulsive nature with each other. What happens? Scattering takes place, scattering takes place at very small angles right.

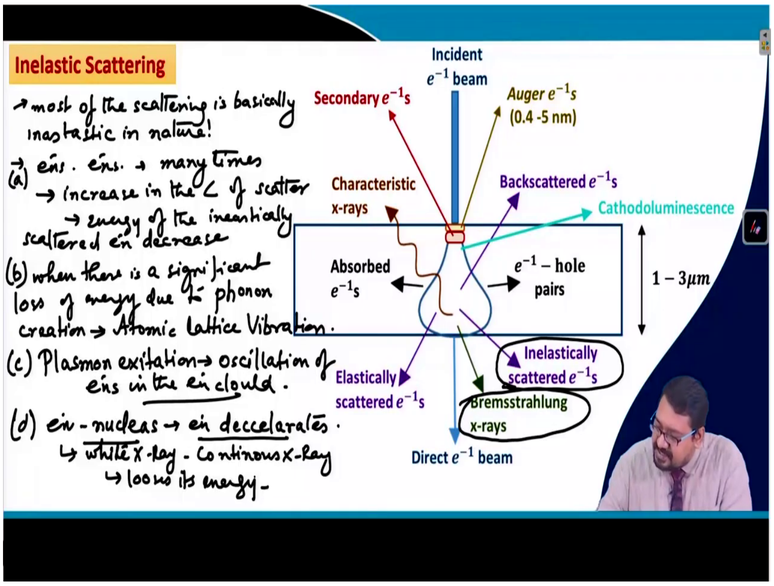
Now, how this is happening? Let us draw, there is a small nucleus and surrounding that are the various K L M N shells of electron where the electrons are there right and few electrons are there in terms of electron cloud in the metal because that any metal or alloy. The metal atoms are not in the ground state, but in a state slightly excited state where the electron of the conducting bands are basically outside and they are basically in form of an electron cloud making the the rest of the atom slightly positive and a slightly higher energy than the non excited state ok 0 state.

So, when the electron falls it is diffracted at a very small angle creating an elastic scattering of electron, which is shown here right. Now on the other hand the electrons sometimes get very close to nucleus and then they scatter by the influence of the nucleus because electrons and nucleus they attract to each other right. So, when the electron basically ok let me change the color of the electron beam here.

So, when the electron basically elect from the electron beam is falling very close to the nucleus and because of its high speed and it is it has the attractive nature of with the nucleus. Because nucleus is positive and the electron is negative what happens that large scattering takes place, the scattering takes place at very large angles right and this is known as elastically back scattered electron ok.

So, elastically back scattered electron or the elastically forward scattered electrons so, this is elastically backscattered electrons. So, both this elastic scattering are without losing detectable amount of energy and therefore, called elastics scattering. So, this is all about elastic scattering.

(Refer Slide Time: 19:44)



Now, that none of the electron beam that scattered is fully elastic in nature. The loss of energy; however, in elastic scattering is not detectable right, but that most of the scattering is basically inelastic in nature ok.

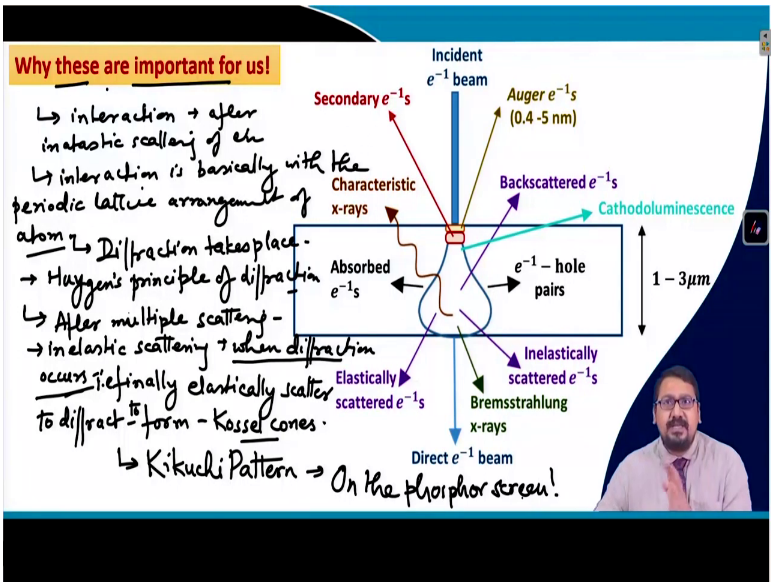
Now, inelastic scattering occurs when electrons from the electron beam interacts with electrons or in the metal or electron cloud many times and while this occurs, there is an increase in the angle of scatter. So, as the electron beams falls and it interacts with electrons in the electron cloud of the metallic surface it basically scattered by one electron cloud one electron.

And then it scatter again and then it scatter again and while it is scattering that every time it can be an elastic scattering, but because there is a loss of energy, which is non detectable in nature. But when the scattering becomes multiple time then the loss of energy becomes detectable in nature and then it becomes an inelastic scattering. And because when multiple scattering takes place there is an increase in the angle of scattering with respect to the incident beam and thereby as the angle of scattering is increased the the energy of the inelastically scattered electron decreases right.

Here is the inelastically scattered electron. So on the other hand so if we say that ok this is 1 and then the second is b. So, well elastic scattering also occurs when there is a significant loss of energy due to phonon creation and what is phonon creation. Phonon creation is atomic lattice vibration. On the other hand inelastic scattering also takes place because of plasmon excitation and what is plasmon excitation, the plasmon excitation is oscillation of electrons in the electron cloud right. So, it increases a lot of oscillation of electrons in the metal present in the electron cloud which leads to inelastic scattering.

The fourth thing is electron interacts with nucleus and it will interact with nucleus always and the in this time what happens that the electron deccelarates and while the electrons deccelarates. It creates white radiation white x-ray radiation or we call it continuous x - rays or we call it bremsstrahlung x - rays and while it radiates bremsstrahlung, it loses its energy and becomes inelastic in nature right. So, these are the four ways by which inelastic scattering takes place.

(Refer Slide Time: 25:34)



Now, we should understand that why we are talking about this and why it is important for us, this is important for us because that after this scattering of electrons because of the interaction with the electrons and the nucleus. So, either it is elastic scattering or inelastic scattering and most of the time it is inelastic scattering.

The interaction ok interaction after this inelastic scattering of electron, this interaction is basically with the periodic lattice arrangement right arrangement of atom inside this metal right.

So, when the interaction is with the periodic lattice arrangement of atom what happens is diffraction takes place and this diffraction is basically following the what you call it the Huygens - Fresnel's principle right, Huygens principle of diffraction. Now as after multiple scattering that is inelastic scattering when diffraction takes place when diffraction occurs it occurs because of the periodic presence of periodic elasticity arrangement in this crystal structure in this particular crystal structure which is made up of atom right.

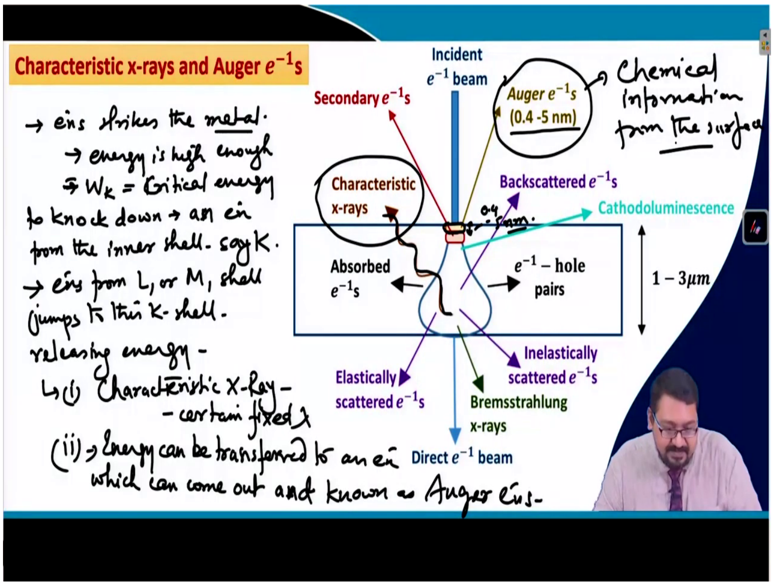
So, diffraction is nothing as we said earlier that it is reinforced scattering where at certain Braggs law following a Laue's equation the at certain angles which should satisfies the Braggs law has a higher intensity and also it must means follow the structure factors rule right and even in this case even in case of electrons. So, the most of the electron beam which has undergone inelastic scattering.

Now, finally, when diffraction occurs that is finally, elastically scattered scatter to diffract to form Kossel cone. Now what does it mean? It means that when electrons scatters multiple time it becomes inelastic scattering. And when it becomes inelastic scattering and multiple scattering takes place the electron seems to come from all possible directions and it seems to come from inside this metal and when it comes from inside this metal it is from all possible directions in space and in 360 degrees. Then what happens? That when the final elastic scattering takes place a single scattering the ones which follows the Braggs principle comes out in a form for a particular HKL plane. It comes out in all possible directions to form a Kossel cone to form a kind of a cone which is called a Kossel cone right and this occurs for each and every possible HKL planes.

And these Kossel cones and this diffraction occurs for a single single HKL plane in both the directions, because in case of electron beam the Braggs diffraction condition is also relaxed. Therefore, instead of n lambda equal to 2 d sin theta, the theta is so small that it can be written as n lambda equal to 2 d theta and theta is in radians not in degrees here.

So, that when we can write that theta then what happens that the theta is so small in a range of say 0.15 degrees and then you think that how much radians it will be. And so when the planes are basically parallel almost parallel to the electron beam the planes basically diffract from both the directions. And therefore, it forms two Kossel cones and therefore, a kind of or structure which is known as a band type structure which is known as kikuchi pattern on the phosphor screen ok. So, this is what is; importance of the elastic and inelastic scattering.

(Refer Slide Time: 32:02)



But if if we look closely there are other radiations that comes out of this material and two important radiations are characteristics x-rays and Auger electrons.

Now we have talked about this earlier in the x-rays that when electron beam is targeted on an anode in a high vacuum chamber it produces x-rays right. First it produces continuous x -rays and when the electrons voltage that is the energy of the electrons is kept on increasing at certain point when the energy reaches a critical energy that is WK we said in the previous lectures.

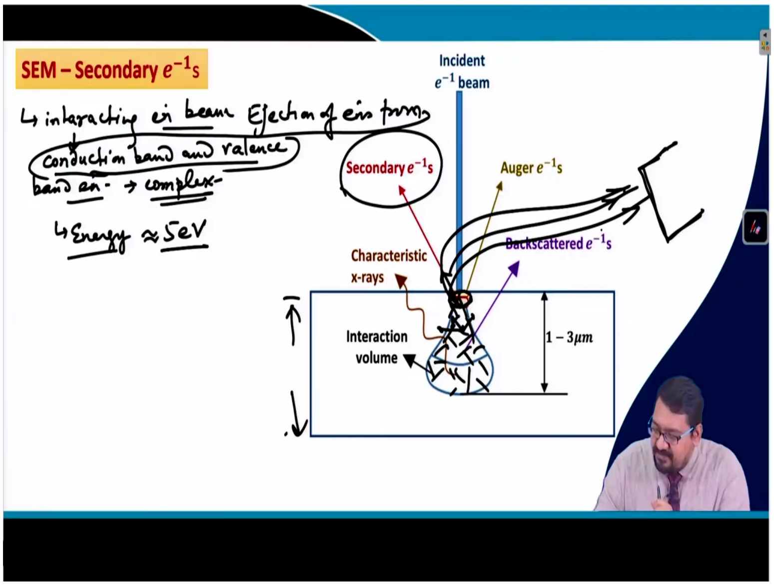
Then K alpha radiations; that is characteristic radiation starts to pop up right. And what is the process of this? Is the same process happens here in the electron microscope inside the TEM the high vacuum is created that is well known. When the electron beam strikes on this thin sample, the thin sample becomes anode right. When it strikes with very high-intensified high energy electron beam when it strikes what happens that the electrons energy can now knock out a lower shell electron say a K shell electron. And this knock down K shell electron goes out and then from the electron from L shell or the M shell full falls into the K shell releasing energy in form of a characteristics x-ray radiation. Or it can give this energy to another electron which can come off out of the sample in form of a roger sorry in form of an Auger electron.

Now, the Auger electron the characteristics x-ray can come off of the sample from quite a distance from inside the sample as shown here. So, it can come off the sample here, but the Auger electron can only come up of the sample because this energy in the Auger electron is quite less and very low very low than even 5 electron volt or lower than that 3 electron or 2 electron volt.

And it can come from the surface of the sample which with the thickness of say 0.4 to 5 nanometer ok. So, it can come from this area of the sample which is have a thickness of 0.4 to 5 nanometer. So, if I write down that when electrons basically strikes the metal and its energy is high enough to so that it becomes equal to the critical energy to knock down.

An electron from the inner shell say K in this case and the electrons from L or M shell jumps to this K shell releasing energy. And this releasing energy could be in form of characteristic x-ray means of a certain fixed lambda or the energy can be transferred to an electron which can come up out as and known as and known as Auger electron ok and the Auger electron has a very low energy. Therefore, it can come off the only from the surface. This Auger electron can be used to carry out chemical information from the surface and it is sometimes used in Auger electron spectroscopy and other applications.

(Refer Slide Time: 37:33)



So, that that is all about Auger electrons, now transmission electron microscope is one equipment where all possible kinds of radiations could be observed, but that few of the radiations like the secondary electrons or the Auger electrons or the the characteristics x - rays or backscattered electrons are usually not observed in the transmission electron microscopy.

They are basically observed in the scanning electron microscopy; where the sample thickness is quite high where the sample thickness is basically very high in sometimes almost half a centimeter or 1 centimeter could be ok only thing is that large samples may not be able to put inside this scanning electron chamber. But the interaction volume where the electron beam falls in it and it scatters in all the direction and it scatters multiple direction ok.

And then it goes the interaction volume with multiple scattering that occurs is limited here and is the same as in case of the transmission electron microscope 1 to 3 micron for different metals or elements having different atomic number. So, higher the atomic number the interaction volume is smaller the lower, the atomic number the interaction volume is higher for the same energy electron beam falling on it.

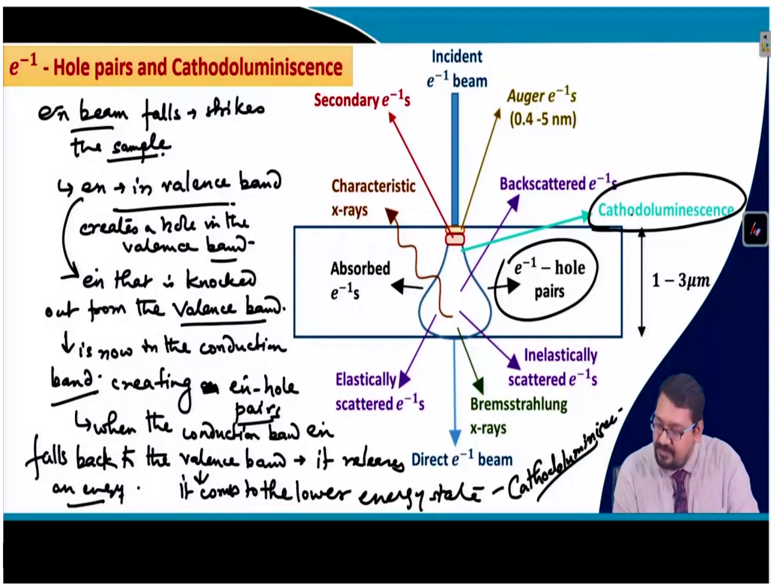
So, one of the very important signals that we obtain in scanning electron microscope is the secondary electrons and secondary electrons are basically used in scanning electron microscope to observe the surface of the material to observe that how the microstructural morphology of the material is. And how it is formed, when the interacting electron beam when the electron beam basically interacts with the conduction band and valence band electrons. And this interaction is quite complex, but what happens that the interaction leads to eject few electrons from the conduction band or the valence band. So, it helps in ejection of electrons from the conduction band and the valence band. Now, this electrons that are ejected from the conduction and valence band have an energy on an average it can vary from 0 electron volt to 50 electron volt.

But on an average the energy of this electrons that is emitted are in a range of about 5 electron volt and the majority of the electrons that are ejected are in this range and therefore, these electrons it has low energy can only come out again from the which are present in the surface of the material.

So, those electrons which have which are coming from the lower of the surface could not be ejected out of the surface, but those which are at the surface right sorry which are at the surface could be ejected out and it has a low energy. And so the detector having a bias is used to attract all these secondary electrons towards the detector.

So, a detector is somewhere placed here and this detector is having in a bias that whatever secondary electron is forming can go to this detector to have and give the information about the microstructural morphology from that position right. So, secondary electron is also very important.

(Refer Slide Time: 42:20)



Finally, in case of TEM and even in case of SEM cathodoluminiscence is produced and when the electron beam falls or strikes the the sample, the electron beam those have less energy comparatively less energy which has less energy not enough energy to knock out the K shell electron to get the characteristic radiation or Auger electrons those can strike.

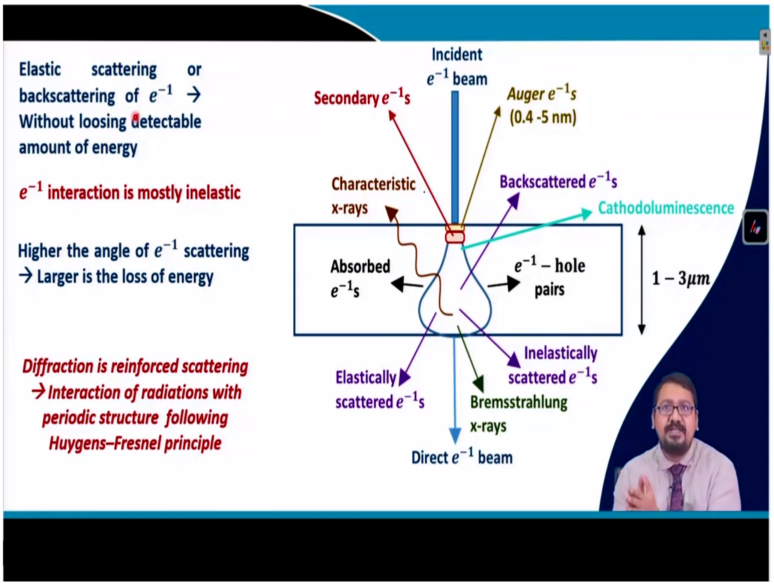
And strike the electron in the valence band and when it strikes the electrons in the valence band it is easier to knock out electron from the valence band right. So, it creates a hole in a valence band. So, it creates a hole in the valence band. The electron that is knocked out from the valence band is now in the conduction band right, creating a an electron hole pair and like that many electron hole pairs. So, creating electron hole pair's right.

And we have shown this here and then when the conduction band electron falls back to the valence band it releases an energy right, it releases an energy because it comes to a lower energy state from the conduction to the valence band right.

So, it comes to the lower energy state releasing an energy which is basically known as cathodoluminiscence ok.

This cathodoluminescence have an wavelength which is much higher than the wavelength of the x- rays and it is maybe sometimes less than the feasible range, but it is even higher than the x- rays ok.

(Refer Slide Time: 46:00)

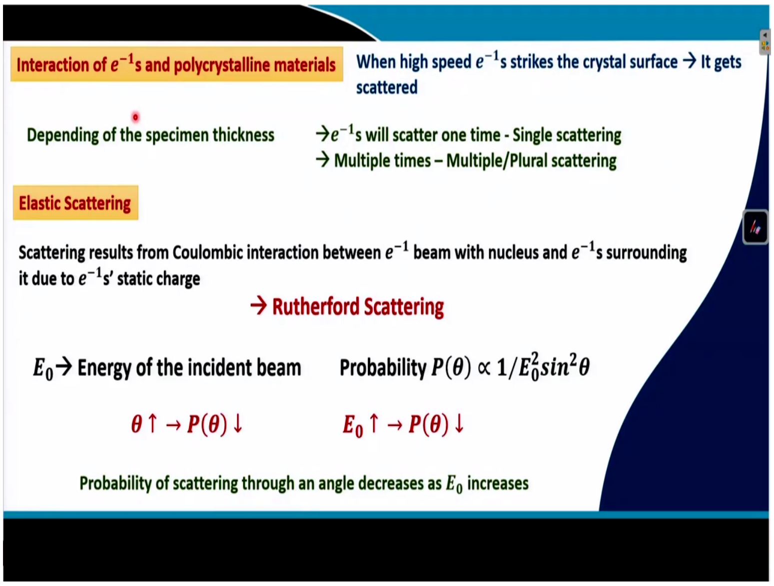


So, what we learn here is that; the electron beam when strikes the metal surface it gives elastic scattering or even it gives elastics back scattering the elastic scattering occurs because the interaction with cloud electron the back scattering occurs because the interaction with the nucleus and it does not lose a detectable amount of energy.

However, when multiple scattering takes place one after another then the energy of the electron that is lost becomes detectable and then this detectable energy is then this then it is called inelastic scattering right. Now one important thing that we learned here is that; higher the angle of the scattering the lower will be the energy of the electron and the as the number of scattering increases the loss of the energy increases.

So, diffraction on the other hand is basically reinforced scattering because of the interaction of this striking electron in the electron beam with the periodic nature of the structure of the sample right and following the Huygens - Fresnel's principle right. And this leads to the production of the Kossel Cone and thereby the kikuchi pattern.

(Refer Slide Time: 47:30)

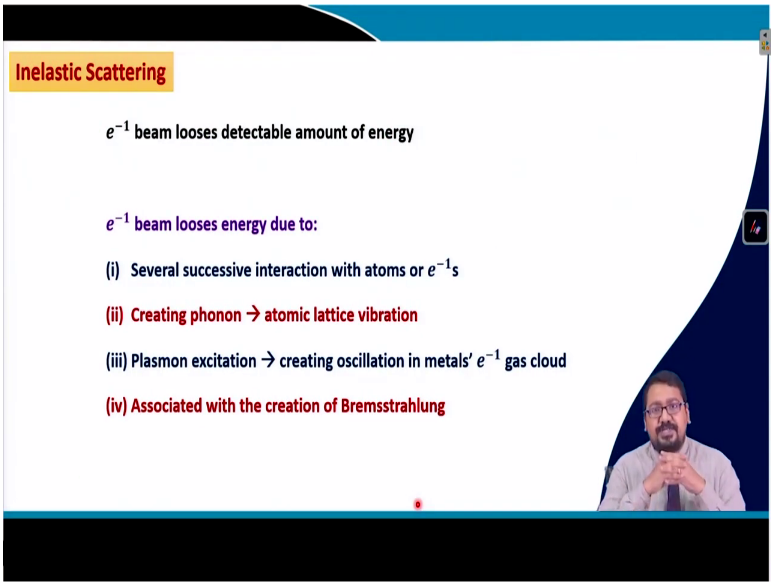


So, what we learned is; interaction of electron with poly crystalline material basically a crystal in a poly crystalline material at very high speed get it self-scattered. And it depending upon the thickness of the specimen the scatter can means the scatter can occur one time which is known as single scattering or it can occur multiple time which known as multiple scattering or plural scattering.

A single scattering will always remain elastic because the loss of energy will be undetectable multiple scattering will be inelastic because there will be a detectable loss of energy. The elastic scattering is basically a coulombic scattering because of the presence of electron cloud and the nucleus and is the same like the Rutherford scattering principle right.

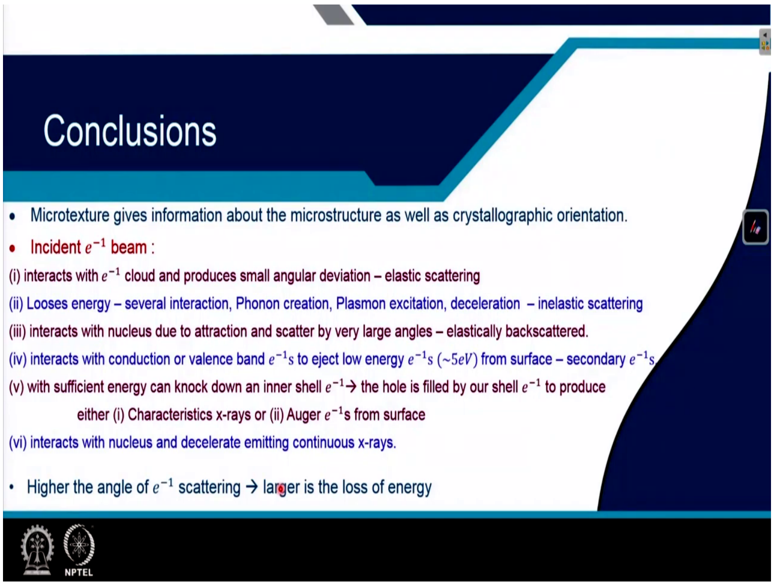
So, the energy of the incident beam basically decreases as the angle of the scattering increases it is given by the probability function P theta which is inversely proportional to the energy square sin square theta. So, as the theta is increasing the probability of the incident sorry the diffracted the scattered beam to going to that theta is decreasing at the same time the energy is increasing as the theta is decreasing. So, the probability of scattering through an angle decreases as the energy increases.

(Refer Slide Time: 49:31)



Inelastic scattering, we have talked in detail about inelastic scattering in the earlier slide, but just to give the summary that inelastic in inelastic scattering the electron beam basically loses a detectable amount of energy. The energy is lost because successive interaction of the electrons with the atom because of phonon creation, because of plasmon excitation and it could be associated with the formation of white rays or characteristic radiations.

(Refer Slide Time: 50:10)



So, finally, we can conclude that; one microstructure gives information mic sorry micro texture gives information about the microstructure as well as the crystallographic orientation. The incident electron beam interacts with the electron cloud and the nucleus to produce small deviation for the electron beam and large deviation for the nucleus and both would be elastic scattering the interaction with nucleus it will be elastically backscattered right.

It loses its energy due to several interaction with for interaction with the electron cloud for formation of phonon, formation of plasmon and formation of continuous x- rays leading to inelastic scattering. It interacts with the conduction and the valence band and ejects a low energy electron which is having an energy of 5 electron volt and is known as secondary electrons mostly it is useful for scanning electron microscopy.

On the other hand, if the energy is sufficient enough, but is lower than to produce the characteristic x-ray as I said it produce a electron hole paired because it knocks down an electron from the valence electron shell. Then when the electron falls on the what you called the from the conduction band to the valence electron shell it produces cathodoluminiscence.

Finally, when sufficient energy is present in the electron beam falling it can knock down a K shell electron and then electron from the L shell goes to the K shell electron to produce either characteristic radiation or the Auger electrons. Interaction of the electron with the nucleus also produces continuous x- rays.

So, finally, the last conclusion is that; higher the angle of scattering which occurs due to multiple scattering there is a higher loss of energy in inelastic scattering.

Thank you very much.